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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT) PCI

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(51) International Patent Classification 6:		(11) International Publication Number: WO 97/12945
C09D 5/04, 7/12	Ŧ	(43) International Publication Date: 10 April 1997 (10.04.97)
(21) International Application Number: PCT/US	396/1594	(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, C7, DF, DK, FR, ES, FI, GB, GE, HU.
(22) International Filing Date: 4 October 1996 (04.10.96)	(04.10.96	IL, IS, IP, KB, KB, KR, KR, LZ, LC, LK, LB, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO,
(30) Priority Dates: 6 October 1995 (06.10.95)		RU, SD, SR, SG, SI, SK, TI, TM, TR, TT, UA, UG, UZ, VI, ARPO patent (TR, LS, MW, SD, SZ, UG), Buratan patent (AM, AZ, BY, KG, KZ, MD, RU, TI, TM), Buropean (A, LD, LD, CAT, ND, NY, RY, RY, RY, RY, RY, RY, RY, RY, RY, R

BB, BG, BR, GB, GE, HU, C, LS, LT, LU, PL, PT, RO, UA, UG, UZ, UO), Eurasian VN, ARPO paten (KE, 1.5, MW, SD, SZ, UG), Burnslan patent (AM, AZ, BY, KG, KZ, MD, RU, TI, ThG), Buropean patent (AT, BE, CH, DE, DK, ES, FT, RC, GB, GR, IE, IT, LU, MC, RL, YT, SB), OAPI patent (BF, BI, CF, CG, CH, GA, GA, MR, NE, SN, TD, TG). S (71) Applicant: CABOT CORPORATION (US/US); 75 State Street, Boston, MA 02109-1806 (US).

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With international search report.
Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of

# (54) THAC: AQUEOUS THIXOTROPES FOR WATERBORNE SYSTEMS

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#### (57) Abstract

Aqueous thisotropes for waterbonne systems are disclosed including an aqueous dispersion of funed silkes and a waterbonne resin. The waterbonne resis is abelieded to the about the group consisting of allyof, acrylds, polyester, polyester, polyester, cocyal, formaldelyde, viayl, and mixtures thereof. The furned silkes has a surface area between about 55 m2/8 and about 410 m3/g, and is present in the system in an amount between about 0.5 and about 10.0 % by weight, of total resis solids.

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TITLE

# AQUEOUS THIXOTROPES FOR WATERBORNE SYSTEMS

# BACKGROUND OF THE INVENTION

## Field of Invention:

The present invention relates to aqueous thixotropes for waterborne systems and, more particularly, to aqueous dispersions of fumed silica for use as effective thixotropes and rheology control agents in waterborne systems.

# Description of the Related Art:

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Both hydrophilic and hydrophobic furned silicas are widely used in the coating industry to improve rheology, for flow control and storage stability, as well as serve as an anti-settling agent for pigments and fillers. The production of hydrophilic furned silica is a well known process. Grades vary in particle and aggregate size. Hydrophobic silica can be produced by treating a furned silica with a suitable agent which will vary depending on the desired degree of hydrophobicity and other characteristics. Such treating agents include, for example, olydimethylsiloxane oils of various molecular weights, dimethyldichlorosilane, clisilazanes, such as hexamethyldisilazane (HMDZ), and mixtures thereof.

In aqueous systems, which include both emulsions and water reducible vehicles, untreated (hydrophilic) silicas are typically not effective because of the large concentrations of silica that are necessary to realize the desired thickening. However, methods exist for increasing the thickening capability of a given concentration of silica by using certain substances as additives to modify the nature of the system. For example, in systems that are not readily responsive to modify the because of inherent chemical properties, the correct additive can often facilitate efficient viscosity and thixotropic control. For example, in highly hydrogen-bonding liquids, the additives that are typically most useful in improving the thickening and thixotropic efficiency of furned silica are cationic surfactants. The surfactants modify and partially impede the interaction between furned silica and the solvent, thereby allowing the furned silica network structure to

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- 2 -

develop, thus resulting in improved viscosity, thixotropy, and suspending properties. In non-hydrogen-bonding liquids, the additives which prove most useful are short chain molecules with more than one functional group capable of hydrogen bonding, such as glycols. These molecules act as bridging compounds between the surface hydroxyls of fumed silica aggregates, which form chains consisting of alternating silica aggregates and organic molecules. Such systems require the separate addition of certain additives to untreated silica dispersions.

As environmental awareness increases, manufacturers face increased pressure to replace conventional solvent based systems. As a result, aqueous systems are increasingly used in many applications such as automotive and industrial coatings, paints, inks, adhesives, and the like.

While hydrophilic and hydrophobic silicas have both been used in solvent-based coating formulations on a commercial scale, their use in aqueous formulations have been plagued with disadvantages. For example, in aqueous systems, either the silica additive must be increased to unacceptable levels or the formulation does not attain the desired level of performance. A demand, therefore, exists for aqueous systems which perform comparably to solvent based systems and, accordingly, for improved additives or methods to accomplish such results.

It is therefore an object of the present invention to provide a versatile and efficient rheological additive for waterborne systems, such as in coatings and other industrial applications. A further object is to provide an additive that fosters stability in aqueous compositions. A still further object is to provide a thixotrope which alleviates many of the waterborne coating rheology control formulating difficulties that exist with other inorganic and organic materials.

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# SUMMARY OF THE INVENTION

Accordingly, the present invention is directed to an aqueous dispersion of furned silica that provides enhanced rheology control and thixotropy to waterborne systems. The furned silica dispersion is uniformly dispersed in the waterborne system such that an amount ranging between 0.5% and 10.0%, by weight, of silica is present in the final composition. The waterborne system, therefore, includes an aqueous dispersion of furned silica, and a waterborne resin. The resin is selected from the group consisting of alkyd, acrylic, polyester, polyether, silicate, urethane, epoxy, formaldehyde, vinyl, and mixtures thereof. The furned silica has a surface area between about 85 m²/g and about 410 m²/g.

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BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a graph of the rheological performance of an epoxy resin system incorporating the present fumed silica dispersion;

FIG. 2 is a graph of the rheological performance of an bisphenol-A epoxy resin system incorporating the present fumed silica dispersion;

FIG. 3 is a graph of the rheological performance of an epichlorohydrin and bisphenol-A resin system incorporating the present fumed silica dispersion;

FIG. 4 is a graph of the rheological performance of a urethane modified epoxy resin system incorporating the present fumed silica dispersion; and

FIG. 5 is a graph of the rheological performance of an acrylic resin system incorporating IG. 6 is a graph of the response of the present fumed silica dispersion in a typical water the present fumed silica dispersion; and reducing alkyd coating formulation.

# DETAILED DESCRIPTION OF THE INVENTION

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which provides enhanced rheology control and thix otropy to aqueous or waterborne systems. The in waterborne systems such as coatings, the present aqueous dispersion of fumed silica will not The present invention is directed to an aqueous dispersion of hydrophilic fumed silica present aqueous dispersion of fumed silica is effective in alkyd, acrylic, polyester, polyether, silicate, urethane, epoxy, formaldehyde, vinyl, and mixtures or modifications thereof as vehicle systems. It has been found that unlike other organic and inorganic materials used as thixotropes typically react with additives in the formulation to produce unexpected side effects after product 12

Fumed silicas useful in this invention are generally characterized by a chain-like structure typically referred to as primary particles, fuse with one another by undergoing collisions at their contact points to form branched, three dimensional chain-like aggregates. The force necessary having high surface area per unit weight. The production of fumed silica is a well-documented process which involves the hydrolysis of silicon tetrachloride vapor in a flame of hydrogen and oxygen. Molten particles of roughly spherical shapes are formed in the combustion process, the diameters of which are varied through process parameters. These molten spheres of fumed silica.

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WO 97/12945

PCT/US96/15940

During cooling and collecting, the aggregates undergo further collision that may result in some to break aggregates is considerable and often considered irreversible because of the fusion. mechanical entanglement to form agglomerates. Compared to the aggregates where the primary particles are fused together, agglomerates are thought to be loosely held together by Van der Waals forces and can be reversed, i.e. de-agglomerated, by proper dispersion in suitable media

about 410 m<sup>2</sup>/g. In the present invention, the fumed silica is preferably in a range from about Cab-O-Sil Division of Cabot Corporation, Tuscola, IL, having a surface area of about 200 m2/g determine the surface area. The surface area of the fumed silica, as measured by the nitrogen adsorption method of S. Brunauer, P. H. Emmet, and I. Teller, J. Am. Chemical Society, Volume 60, Page 309 (1938) and commonly referred to as BET, typically ranges from about 85 m²/g to 175 m²/g to about 225 m²/g, and are of a high purity. High purity means that the total impurity content is typically less than 1% and preferably less than 0.01% (i.e., 100 ppm). Although many commercially available fumed silicas are suitable, CAB-O-SIL® fumed silica, available from the is most preferred. Such a silica has been found to be of high quality and is readily dispersable. The size of the primary spherical particles that comprise the fumed silica aggregate 9

present invention has a pH ranging between 7.0 and 9.5. The fumed silica dispersion of the present invention is preferably prepared by the method described by Miller et al., in U.S. Patent The fumed silica of the present invention is uniformly dispersed in a stable aqueous medium (e.g. deionized water) using conventional methods known to those skilled in the art. By uniformly dispersed is meant that the aggregates are isolated and well distributed throughout the potassium hydroxide, ammonia and the like. Preferably, the fumed silica dispersion of the aqueous fumed silica dispersions, available from the Cab-O-Sil Division of Cabot Corporation, No. 5,246,624, the disclosure of which is incorporated herein in its entirety by reference. and 10.5 and may be adjusted by the addition of a suitable base such as sodium hydroxide, Although many commercially available fumed silica dispersions are suitable, CAB-O-SPERSE® medium. By stable is typically meant that the aggregates will not re-agglomerate and settle ou (e.g. form a hard, dense sediment). The fumed silica dispersion should have a pH between S Tuscola, IL, are most preferred The waterborne systems of the present invention are prepared by combining or mixing the aqueous dispersions of fumed silica directly with a waterborne resin, such as an alkyd, acrylic,

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PCT/US96/15940

polyester, silicate, urethane, epoxy, and the like, or with a formulation containing a waterborne resin under low shear conditions (i.e., to prevent foaming) until a uniform homogeneous composition is obtained. Typically, the fumed silica dispersions range from about 10% to about 45% solids, by weight; and, preferably, between 15% and 30% solids, by weight. Most referably, a fumed silica dispersion of about 20% solids, by weight, has been found to maximize the toading level of silica while maintaining the colloidal stability of the dispersion.

The aqueous dispersions of fumed silica of the present invention are useful in aqueous systems to provide effective rheological control (i.e., viscosity and thixotropy) for example, in industrial and automotive coating, adhesive, paint, and ink applications. It has been found that another advantage of using an aqueous dispersion of fumed silica in waterborne resin systems is to provide rheology and sag-control, and anti-settling. It is also believed that a stable matrix is formed in waterborne formulations after the incorporation of the fumed silica dispersion due to electrostatic interaction. During the high-shear processes usually found in product application, the matrix disintegrates, thereby reducing viscosity and permitting near-Newtonian flow. After application, the inorganic matrix reforms at a predictable rate to provide resistance to sagging and edge-pull during film coalescence and/or cure. This matrix remains unmodified through the many physical film changes during conversion from liquid to solid, thus providing a predictable application consistency.

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The aqueous dispersion of furned silica has been found to be an effective suspension agent in some formulations due to its internally-generated structure. The product forms a matrix capable of stopping or significantly retarding undesirable striation and pigment settling, even in products containing powdered zinc or other unusually heavy pigments. It is noted that the addition of dry hydrophilic furned silica to waterborne systems has been found to be ineffective as a rheology control agent. In particular, the viscosity of the system will tend to continually increase over time, thereby not achieving stability. In addition, the dry silica is difficult to handle and disperse, and may tend to settle out at higher loading levels.

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Other advantages of the present dispersions are that they will not migrate in wet or dry films and are unaffected by heat and atmospheric exposure in the dried/cured film. The dispersion is inherently non-yellowing and will not contribute to color changes or drift in either

WO 97/12945

PCT/US96/15940

the wet or dry state. Moreover, the present aqueous dispersions of fumed silica are biologically inactive and is not expected support microbial activity.

The present aqueous dispersions of fumed silica used as aqueous thixotropes in water based systems are responsive to changes in pH, but have been found to be effective in the 7.0 to 9.5 pH range commonly used in products formulated for industrial and commercial use. The present dispersions have also been found to be effective in higher pH ranges, depending specifically upon the individual formulation.

Although the loading level, as a percent of total or resin solids, the precise method of incorporation, and the stage of manufacture at which this is accomplished, all play a significant role in the final effectiveness of the dispersion, the aqueous dispersion of fumed silica of the present invention has been shown to be effective in many systems used in formulating waterborne products.

The present invention will be further illustrated by the following examples, which are intended to be illustrative in nature and are not to be considered as limiting the scope of the invention.

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### EXAMPLE I

An aqueous colloidal dispersion of fumed silica, which can be used as an aqueous thixotrope in the present invention, was prepared and evaluated as follows.

Approximately 205.24 grams of deionized water and 0.4 gram of 38% hydrochloric acid
were added to a commercial Waring blender. While mixing, 80 grams of CAB-O-SIL® PTG
grade fumed silica were added to the blender. When the addition was complete, the mixture was
stirred at a high rate for about five minutes. After the stirring was completed, about 108.52
grams of water were added to the mixture followed by about 6.12 grams of a 10% potassium
hydroxide solution stabilizer. After the addition of the stabilizer, the mixture was stirred for an
additional two minutes. The aqueous fumed silica dispersion prepared had a 20% solids level,
a viscosity of about 65 centipoise, and a pH of 9.

PCT/US96/15940

### **EXAMPLE II**

A scaled-up volume of the dispersion of EXAMPLE I was prepared, using 2-amino-2-methyl-1-propanol, available as AMP-95<sup>rd</sup> from Angus Chemical Company, Buffalo Grove, IL, as the base-stabilizer. Approximately 1283.80 grams of water were mixed with 0.251 gram of 38 % hydrochloric acid. About 499.92 grams of CAB-O-SIL.® PTG grade fumed silica were then added to the water/acid mixture, and was stirred for about 20 minutes. 678 grams of deionized water was then added to the mixture, followed by 10.54 grams of the AMP-95<sup>rd</sup> stabilizer. The aqueous fumed silica dispersion prepared had a 20.22% solids level, and a pH of 9.

### **EXAMPLE III**

dispersion prepared in EXAMPLE I) and a waterborne epoxy resin, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope for waterborne systems. The aqueous funed silica dispersion was mixed with Waterpoxy® 701 Resin epoxy curing agent, available from Henkel Corporation, Ambler, PA, until a loading level of 2% dry silica on resin solids was achieved. The viscosity (centipoise), shear thinning index (STI), and pH were then measured after sample preparation (0 days), and after periods of 1 and 7 days. The viscosity was measured on a Brookfield viscometer using Nos. 3 or 4 spindle at 6 and 60 RPM's. The experimental results are presented below in Table 1.

#### TABLE 1

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DAYS	0	-	7 : T
VISCOSITY			
Sample 1, 6 RPM	1500	1500	1500
Sample 1, 60 RPM	1442	1442	1442
Sample 2, 6 RPM	3300	3900	3800
Sample 2, 60 RPM	1990	2540	2590

WO 97/12945

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		and the state of t			
	STI				
	Sample 1	1.04	1.04	1.04	
	Sample 2	1.66	1.53	1.51	,
	Hd			·	
5	Sample 1	11.33	11.33	11.33	
	Sample 2	11.29	11.41	10.99	

FIG. 1 is a graph of the viscosity of a control sample (Sample 1), a waterborne epoxy resin without a rheology control agent, versus the present waterborne system (Sample 2) over time. Both Table 1 and FIG. 1 illustrate that the present waterborne system achieved stable performance after about 1 day and a desired increase in viscosity.

### **EXAMPLE 1Y**

A waterborne system, including an aqueous fumed silica dispersion (similar to the dispersion prepared in EXAMPLE I) and a waterborne bisphenol-A epoxy resin, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope for waterborne systems. The aqueous fumed silica dispersion was mixed with EPI-REZ® 3510-W-60 nonionic, aqueous dispersion of bisphenol-A epoxy resin, available from Shell Chemical Co Houston, TX, until a loading level of 2% dry silica on resin solids was achieved. The viscosity (centipoise), shear thinning index (STI), and pH were then measured after sample preparation (0 days), and after periods of 1 and 7 days. The viscosity was measured on a Brookfield viscometer using Nos. 3 or 4 spindle at 6 and 60 RPM's. The experimental results are presented below in

#### TABLE 2

DAYS	0	-	Ĺ
VISCOSITY			

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WQ 97/12945

PCT/US96/

7.04	7.04	5.70	Sample 2
3.41	3.41	3.41	Sample 1
			pH
1.85	1.85	1.97	Sample 2
1.24	1.24	1.24	Sample 1
		,	STI
260	270	264	Sample 2, 60 RPM
400	500	520	Sample 2, 6 RPM
178	178	178	Sample 1, 60 RPM
220	220	220	Sample 1, 6 RPM

FIG. 2 is a graph of the viscosity of a control sample (Sample 1), a waterborne bisphenol-A epoxy resin without a rheology control agent, versus the present waterborne system (Sample 2) over time. Both Table 2 and FIG. 2 illustrate that the present waterborne system achieved stable performance, particularly the Sample 2 measured at 60 RPM, and a desired increase in viscosity. The Sample 2 viscosity measured at 6 RPM decreased more rapidly.

### EXAMPLE V

A waterborne system, including an aqueous furned silica dispersion (similar to the dispersion prepared in EXAMPLE I) and a waterborne epichlorohydrin and bisphenol-A epoxy resin, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope for waterborne systems. The aqueous furned silica dispersion was mixed with EPI-REZ® 3510-WY-55 (55% solids) dispersion of Econ™ 1001F condensation product of epichlorohydrin and bisphenol-A in water, available from Shell Chemical Co., Houston, TX, until a loading level of 2% dry silica on resin solids was achieved. The viscosity (centipoise), shear thinning index (STI), and pH were then measured after sample preparation (0 days), and after

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WO 97/12945

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periods of 1 and 7 days. The viscosity was measured on a Brookfield viscometer using Nos. 3 or 4 spindle at 6 and 60 RPM's. The experimental results are presented below in Table 3.

- 10 -

#### TABLE 3

	DAYS	. 0	-	7
S	VISCOSITY			
	Sample 1, 6 RPM	2700	2700	2700
	Sample 1, 60 RPM	1010	1010	1010
	Sample 2, 6 RPM	3000	. 5600	15400
	Sample 2, 60 RPM	1130	1950	3700
0	STI			
	Sample I	2.67	2.67	2.67
	Sample 2	2.65	2.87	4.16
	Hq			
	Sample 1	8.74	8.74	8.74
Ċ,	Sample 2	9.38	9.36	7.40

FIG. 3 is a graph of the viscosity of a control sample (Sample 1), a waterborne epichlorohydrin and bisphenol-A epoxy resin without a rheology control agent, versus the present waterborne system (Sample 2) over time. Both Table 3 and FIG. 3 illustrate that the present waterborne system achieved a desired increase in viscosity while providing semi-stable performance in thixotropy. The Sample 2 viscosity measured 6 RPM increased rapidly. The Sample 2 measured at 60 RPM increased less rapidly and was more stable.

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### EXAMPLE VI

A waterborne system, including an aqueous fumed silica dispersion (similar to the dispersion prepared in EXAMPLE I) and a waterborne urethane modified epoxy resin, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope for waterborne systems. The aqueous fumed silica dispersion was mixed with EPI-REZ® 5520-W-60 nonionic aqueous dispersion of urethane modified epoxy resin, available from Shell Chemical Co., Houston, TX, until a loading level of 2 % dry silica on resin solids was achieved. The viscosity (centipoise), shear thinning index (STI), and pH were then measured after sample preparation (0 days), and after periods of 1 and 7 days. The viscosity was measured on a Brookfield viscometer using Nos. 3 or 4 spindle at 6 and 60 RPM's. The experimental results are presented below in Table 4.

#### TABLE 4

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	<b>-</b>	·	
	2600	2600	2600
Sample 1, 60 RPM	1440	1440	1440
	29450	17200	17900
Sample 2, 60 RPM	3710	2730	2920
	3.89	3.89	3.89
	7.94	6.30	6,13
	3.51	3.51	3.51
	7.37	7.53	7.40

WO 97/12945

- 12 - .

PCT/US96/15940

FIG. 4 is a graph of the viscosity of a control sample (Sample 1), a waterborne urethane modified epoxy resin without a rheology control agent, versus the present waterborne system (Sample 2) over time. Both Table 4 and FIG. 4 illustrate that the present waterborne system achieved stable performance after about 1 day and a desired increase in viscosity.

## EXAMPLE VII

A waterborne system, including an aqueous fumed silica dispersion (similar to the dispersion prepared in EXAMPLE I) and a waterborne acrylic copolymer resin, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope for waterborne systems. The aqueous fumed silica dispersion was mixed with Neocryl<sup>4</sup> A-639 waterborne acrylic copolymer, available from Zeneca Resins, Wilmington, MA, until a loading level of 2% dry silica on resin solids was achieved. The viscosity (centipoise), shear thinning index (STI), and pH were then measured after sample preparation (0 days), and after periods of 1 and 7 days. The viscosity was measured on a Brookfield viscometer using Nos. 3 or 4 spindle at 6 and 60 RPM's. The experimental results are presented below in Table 5.

#### TABLE 5

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	L		480	212	. 580	258		2.26	2.25
		•	480	212	089	282		2.26	2.41
	0		480	212	540	254		2.26	2.13
	DAYS	. VISCOSITY	Sample 1, 6 RPM	Sample 1, 60 RPM	Sample 2, 6 RPM	Sample 2, 60 RPM	ILS	Sample 1	Sample 2
•					Ŕ				

PCT/US96/15940

- 13

 pH
 6.53
 6.53
 6.53

 Sample 1
 6.53
 6.59
 6.49
 6.53

FIG. 5 is a graph of the viscosity of a control sample (Sample 1), a waterborne urethane modified epoxy resin without a rheology control agent, versus the present waterborne system (Sample 2) over time. Both Table 5 and FIG. 5 illustrate that the present waterborne system achieved stable performance after about 1 day and desired increase in viscosity.

## EXAMPLE VIII

A coating formulation, incorporating the aqueous furned silica dispersion of the present invention, a waterborne acrylic resin system, and various additives, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope in coatings. A high gloss waterborne topcoat, based on Neocryl® XK90 acrylic copolymer emulsion, available from Zeneca Resins, Wilmington, MA, was prepared. The coating formulation, as set forth below in Table 6, was prepared by first mixing the components of Part A in a Waring Blender at high speed for about 5 minutes. The Part B components were then added and dispersed into the Part A mixture in the Waring Blender at high speed for about 5 minutes. The viscosity (mPas), STI, and pH were then measured after periods of 2 days, 25 days, and 60 days. These measurements were taken for a high gloss aqueous top coat formulation without any commercial thickening agent (Control), and a similar formulation utilizing an aqueous fumed silica dispersion (similar to the dispersion prepared in Example I) as an aqueous thixotrope (Sample). The viscosity of the coatings was measured on a Brookfield Rotary Viscometer RVD-II using spindle Nos. 1 or 2 at speeds of 0.5, 1.0, 2.5, 5.0, and 10.0 RPM. The STI is a ratio of the measured viscosity at 0.5 and 5.0 RPM's. The experimental results are presented below in Table 7.

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#### TABLE 6

	FORMULATION	
(WT. %)	CONTROL	
(WT: %)	SAMPLE	

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WO 97/12945

PCT/US96/15940

- 14 -

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	NaNO <sub>2</sub> , 30% sol'nrust iṇhibitor	Neocryl®XK90	Butyl Glycol	Part B:	TiO <sub>2</sub>	Butyl Glycol-coalescent	Aqueous Fumed Silica Dispersion (i.e., Example I)	Disperser Ayd W22 dispersant (Daniel)	Dehydran™ 1293 defoamer (Henkel)	Neocryl® XK90 acrylic dispersion	Deionized water	Part A:
100.00	0.41	48.0	1.78		18.78	1.78	1.	0.34	0.30	23.65	4.96	
100.00	0.41	47.10	1.78		18.78	1.77	5.00	0.34	0.30	23.56	0.94	

#### TABLE 7

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Sample, 0.5 RPM	10.0 RPM	5.0 RPM	2.5 RPM	1.0 RPM	Control, 0.5 RPM	VISCOSITY (mPas)	DAYS
2560	60	66	72	90	100		2
1280	52	54	58	60	90		25
1280	55	56.	56	70	.70		60
	2560 1280	10.0 60 52 le, 0.5 RPM 2560 1280	5.0 RPM     66     54       10.0     60     52       e, 0.5 RPM     2560     1280	2.5 RPM     72     58       5.0 RPM     66     54       10.0     60     52       e, 0.5 RPM     2560     1280	1.0 RPM     90     60       2.5 RPM     72     58       5.0 RPM     66     54       10.0     60     52       e, 0.5 RPM     2560     1280	SI, 0.5 RPM     100     90       1.0 RPM     90     60       2.5 RPM     72     58       5.0 RPM     66     54       10.0     60     52       e, 0.5 RPM     2560     1280	ISCOSITY (mPas)     100     90       bi, 0.5 RPM     100     90     60       1.0 RPM     90     60     58       2.5 RPM     66     54     50       10.0     60     52     52       e, 0.5 RPM     2560     1280     1280

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WQ 97/12945

PCT/US96/15940

- 15 -

2.5 RPM	1010	752	784
5.0 RPM	720	620	648
10.0 RPM	. 224	508	536
STI (0.5/5.0RPM)			
Control	1.52	1.66	1.25
Sample	3.56	2.06	1.98

As illustrated above, the aqueous dispersion of the present invention can be incorporated not only in resins but also in coating formulations.

## **EXAMPLE IX**

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finess of grind <10 microns (the graph was converted from mPas to centipoise). As illustrated coating formulation, as set forth below in Table 8, was prepared by mixing the components in a Waring blender at high speed for about 5 minutes. The viscosity (mPas) and STI were then measured after periods of 1, 18 and 60 days. These measurements were taken for an aqueous industrial maintenance coating of formulation with and without a commercial thickening agent (Borchige!" L75N, 54 %), and a similar formulation utilizing an aqueous fumed silica dispersion (similar to the dispersion prepared in Example 1) as an aqueous thixotrope (Sample). The viscosity of the coatings was measured on a Brookfield Rotary Viscometer. The experimental results are presented below in Table 9.. FIG, 6 is a graph illustrating the viscosity aging over time, taken from the data in Table 9, and corresponding to a shear rate of 1.4 dynes/sec. At a in FIG. 6, the aqueous fumed silica dispersion achieved and maintained a stable workable viscosity for an extended period of time. The Control sample exhibited a high initial increase available from Hoechst Celanese, Fine Chemicals Division, Charlotte, NC, was prepared. The A coating formulation, incorporating the aqueous fumed silica dispersion of the present invention, a waterborne alkyd resin system, and various additives, was prepared and evaluated to determine the effectiveness of the present dispersion as a thixotrope in coatings. An aqueous industrial maintenance coating based on Resydrol® AY 466 W, 38%, alkyd resin emulsion, in viscosity and substantial loss over time. 15

WQ 97/12945

PCT/US96/15940

- 16 -

TABLE 8

Į				
	FORMULATION	CONTROL (WT. %)	SAMPLE (WT. %)	
Resy	Resydrol®AY466 W,38%	70.00	.69.02	_
Aque	Aqueous Ammonia, 10%	3.00	2.96	
Addi	Additol VXW 4940 drying agent (Hoechst Celanese)	1.30	1.28	
Titan	Titanox 2300 (Kronos)	21.24	20.94	
Borchige thickener	Borchigel L7SN, 54% thickener	1.86		
A que Disp	Aqueous Fumed Silica Dispersion (i.e., Example I)	1	4.81	
Additol™ defoamer	Additol** VXW 4973 defoamer	0.30	0.30	
Surf	Surfynol SE-F wetting agent (Air Products)	0.30	0.30	
Addi antisl	Additol <sup>14</sup> XL 297 antiskinning agent	0.40	0.39	
Deio	Deionized water	1.60	l	
		100.00	100:00	

TABLE 9

3.472 0.588 3.087 8 0.588 4.340 2.940 8 0.785 6.660 1.985 VISCOSITY Control without DAYS Borchigel" thickener Control Sample STI 20 22

- 17 -

PCT/US96/15940

Control	1.54	1.27	1.66
Control without thickener	3.13	1.81	1.88
Sample ·	3.33	4.64	2.63

As illustrated above, the aqueous dispersion of the present invention can be incorporated not only in resins but also in coating formulations.

Although particular embodiments of the invention have been described in detail for purposes of illustration, various changes and modifications may be made without departing from the scope and spirit of the invention. Accordingly, the invention is not to be limited except as by the appended claims.

What is claimed is:

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WO 97/12945

- 18 -

PCT/US96/15940

;

**CLAIMS** 

A waterborne system, comprising: an aqueous dispersion of fumed silica; and

a waterborne resin;

- wherein said fumed silica has a surface area between about 85 m $^2$ /g and about 410 m $^2$ /g.
- The waterborne system of claim 1, wherein said fumed silica has a surface area between about 175 m²/g and about 225 m²/g.
- The waterborne system of claim 2, wherein said fumed silica has a surface area of about 200 m³/g.
- 4. The waterborne system of claim 1, wherein said fumed silica has an impurity level of less than 1 %.
- The waterborne system of claim 1, wherein said system comprises between about 0.5 and about 10.0% furned silica, by weight, of total resin solids.
- 15 6. The waterborne system of claim 5, wherein said system comprises between about 0.5 and about 5.0% fumed silica, by weight, of total resin solids.
- 7. The waterborne system of claim 6, wherein said system comprises about 2.0% fumed silica, by weight, of total resin solids.
- 8. The waterborne system of claim 1, wherein said aqueous dispersion of fumed silica20 has between 10% and 45%, by weight, silica solids.
- 9. The waterborne system of claim 8, wherein said aqueous dispersion of fumed silica has between 15% and 30%, by weight, silica solids.

ALIBRATION OWN ALIBORATE

The waterborne system of claim 9, wherein said aqueous dispersion of fumed silica

has 20%, by weight, silica solids.

10.

PCT/US96/15940

WQ 97/12945

FIG. 1

DAYS

200

1000

1200

2000

7200

3000

2200

000+

/ISCOSITY(cps)

AUSTOCKIN - WID 8712846A1 | >

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17.6

SAMPLE 1,6 RPM SAMPLE 2,6 RPM SAMPLE 2,6 RPM SAMPLE 2,60 RPM

ς

The waterborne system of claim 11, wherein said agueous dispersion of fumed silica

The waterborne system of claim 1, wherein said aqueous dispersion of fumed silica

has a pH between about 5.0 and about 10.5.

Ξ

has a pH between about 7.0 and about 9.5.

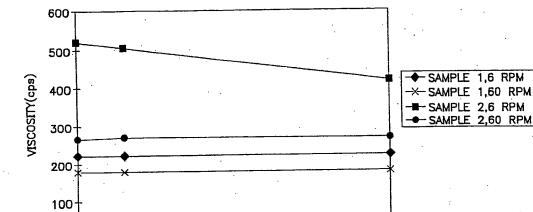
12.

The waterborne system of claim 1, further comprising at least one additive selected from the group consisting of surfactants, coalescing solvents, pigments, defoamers, <u>.</u>

antiskinning agents, drying agents, wetting agents, thickening agents, dispersing agents, biocides, and corrosion inhibitors. 2

The waterborne system of claim 1, wherein said waterborne resin is selected from the group consisting of alkyd, acrylic, polyester, polyether, silicate, urethane, epoxy, formaldehyde, vinyl, and mixtures thereof. ₹.





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FIG. 2

DAYS

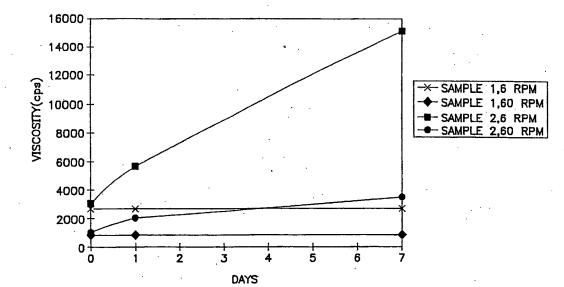


FIG. 3

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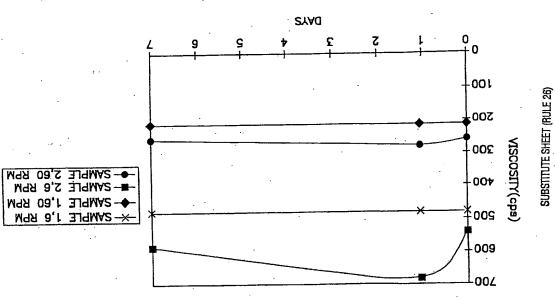
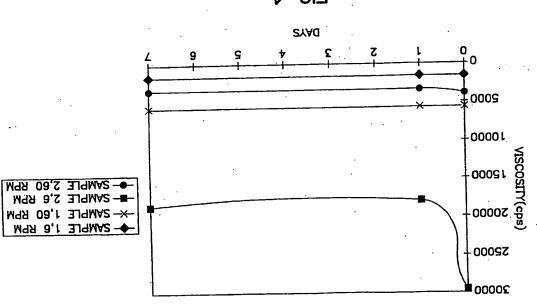
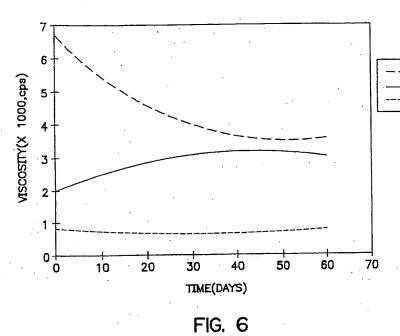


FIG. 4



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NO ADDITIVE(CONTROL) ASSOCIATIVE THICKENER SAMPLE



PCT/US96/15940

INTERNATIONAL SEARCH REPORT

29 January 1997 see abstract
see page 2, line 48-52
see page 3, line 1-5
see page 4, paragraph 3 US 4 455 331 A (ROBERT J. BARSOTTI) 19 June 1984 see claims 1-3 WO 94 18277 A (CABOT CORPORATION) 18 August 1994 see abstract see claim 2 European Patent Office, P.B. Skil Patendaan 2 NL - 2250 HV Rijewiji Tel. (+ 31-70) 340-2040, Tx. 31 651 epo ni, Fax: (+ 31-70) 340-3016 EP 0 399 442 A (BASF CORPORATION) 28 November 1990 ation of box C. PC1/US 96/15940 1 1. 02. 97 1-4,13, 14 Relevant to claim No.

Miller, A

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